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(54) PROCESS FOR THE PRODUCTION OF PERFLUORO HYDROCARBONS

5 (71) We, AKADEMIE DER WISSENSCHAFTEN DER DDR, of 5, Rudower Chaussee, 1199 Berlin, German Democratic Republic, a Corporation organised under the laws of the German Democratic Republic, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The present invention is concerned with a process for the production of perfluoro hydrocarbons from solid fluorinated hydrocarbon polymers and preferably from polytetrafluoroethylene.

15 It is known that perfluorinated macromolecules can be decomposed thermally and by the action of other forms of energy to give stable fluoroalkyl radicals. A varying mixture of different decomposition products, with certain amounts of the starting material, are always obtained. In the case of an attempt to achieve a total conversion, as a rule, complete decomposition occurs, whereby, in addition to coke-like masses, fluorine and various other fluorine-containing decomposition products are formed. On the other hand, the production 20 of low molecular weight perfluoro-hydrocarbons, especially of perfluorinated olefins, is, at the present time, still a laborious synthesis.

25 However, there is an increasing need for low molecular weight, highly reactive products with a high fluorine content for the production of perfluorinated derivatives of which, for example, the perfluorinated carboxylic acids are already of great interest and importance for the production of textile adjuvants, tensides, biologically-active substances and other valuable technical-chemical products.

30 It is also known that, in the case of radiation-chemical reactions, various types of chain breakdown take place which lead

35 to substitution and exchange reactions, no definite products being obtained. It is also known that, under these conditions, the polymer decomposition takes place statistically.

40 Since perfluorinated organic chemicals of varying types with functional groups are of increasing importance for the production of chemical and technical products, for example, textile adjuvants, tensides, lubricants, plastics and rubbers and plasticisers, as well as biologically very active substances, especially biocides and pharmaceuticals, there is an increasing interest for perfluorinated organic chemicals of hitherto known or unknown constitution.

45 The degree of difficulty in the production of these products is best characterised in that anhydrous hydrofluoric acid or elementary fluorine are needed for their synthesis. As a result of the expensive synthesis resulting from the degree of difficulty, these compounds are only employed for very special purposes.

50 It is an object of the present invention to avoid laborious syntheses and thus economically to prepare perfluorinated compounds.

55 The problem forming the basis of the present invention is to provide a process for the production of perfluorinated compounds.

60 According to the present invention, there is provided a process for the production of perfluoro hydrocarbons with a chain length of up to 30 carbon atoms or of substituted derivatives thereof or of corresponding perfluorocarboxylic acids from solid perfluorinated hydrocarbon polymers, wherein a solid perfluorinated hydrocarbon polymer, preferably polytetrafluoroethylene, is irradiated with electron beams, the intermediately formed fission products are optionally reacted with a reactant and the perfluoro hydrocarbons or derivatives thereof or the corresponding perfluoro-

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carboxylic acids thereby formed are immediately removed from the radiation zone, for example by a current of carrier gas. The products obtained can be subsequently fractionated. It is advantageous to use the material to be irradiated and the current of carrier gas at an elevated temperature of up to the temperature of thermal decomposition of the polymer starting material.

The process according to the present invention can be carried out in a device comprising a temperature-controllable, double-walled irradiation vessel for containing the material to be irradiated, a gas inlet and a gas outlet arranged at the height of the reaction zone, a sealing and irradiation-permeable foil and means for regulating the electron density or the degree of decomposition in the irradiation bed. This device preferably has a temperature-controllable sieve base below which is arranged a collecting device and a transport window can be provided below the sieve base.

The irradiated material can be supplemented by fresh polymer in an amount corresponding to the amount of polymer which has been converted. A bed of predetermined thickness of the material to be irradiated is preferably passed under the source of radiation and unreacted or partially reacted material can be recovered and/or recycled. For increasing the decomposition yield, oxygen or a mixture of oxygen with an inert gas can be used as carrier gas. The fission products can be fractionally condensed and/or selectively separated from the carrier gas.

It is advantageous to allow intermediately formed radical fission products from the irradiation-chemical decomposition at temperatures of up to those of thermal decomposition of polytetrafluoroethylene or of analogously fluorinated hydrocarbon polymers to react with suitable added substances, which are preferably also reactive under the selected conditions, to give substituted, perfluoro hydrocarbon derivatives, the reaction products being removed from the irradiation zone immediately after they are formed. This advantageously takes place by means of a current of carrier gas of appropriate temperature, flow velocity and composition.

The separation of the perfluoro hydrocarbon derivatives from the current of carrier gas advantageously takes place according to the known methods of fractional condensation. The reactive substances are either added to the solid polymers before commencement of the irradiation or are added during the irradiation in suitable manner, for example, by blowing in, spraying in or sprinkling in or are contained in the carrier gas or function

simultaneously as carrier gas. A combination of different methods of addition is also possible and, in the same way, two or more reactive substances can be fed in simultaneously or successively and allowed to react in appropriate manner with the intermediately arising radical polymeric fission products.

The process can be carried out continuously or discontinuously and unreacted or partially reacted starting material can be recovered and/or recycled. As reactive substances, those are especially useful which are known to be convertible by electron irradiation into a radical-like or activated state or are themselves radical-like.

It is also of advantage that the products obtained by irradiating fluorinated hydrocarbon polymers with electron beams, optionally in the presence of materials which promote decomposition, are additionally brought into contact and reacted with substances capable of reaction therewith when the products formed are still *in statu nascendi*. According to this embodiment of the process, the substances capable of reaction are not present during the formation of the products by the action of electron beams but rather the products formed and the substances capable of reaction are brought together outside the irradiation zone.

We have also found that it is advantageous to bring together and to react products formed by the action of electron radiations on fluorinated hydrocarbon polymers, which are themselves in an activated state, outside the irradiation zone and during the time in which they are still *in statu nascendi*, with the substances capable of reaction therewith.

In certain cases, it is advantageous, for example when the period of time in which the products formed are *in statu nascendi* is very short or when a mixture of the products formed obtained by the irradiation of fluorinated hydrocarbon polymers is first to be fractionated, again to treat the products formed with electron radiations and then to react them with substances capable of reaction therewith. The process according to the present invention can also undergo a further variation in that the substances capable of reaction are also subjected to the action of electron radiations, not only alone but also together with products formed from the fluorinated hydrocarbon polymers and with the products formed.

The process according to the present invention can also be carried out using, as reaction components, the products formed by the action of electron radiations on fluorinated hydrocarbon polymers in the presence of oxygen or of reactive sub-

- stances. According to an especially advantageous embodiment of the process, the radiation-chemically formed products, i.e. the perfluoro-fission products or compounds already reacted with oxygen or other substances, are, after fractionation, reacted as individual fractions or as groups of fractions with substances capable of reaction therewith.
- We have also found that the products formed can be reacted with polymers, after the fractionation, as individual fractions or as groups of fractions.
- If, during the action of the electron radiations on the fluorinated hydrocarbon polymers, reactive substances are present, for example by adding the reactive substances to the carrier gas current in the form of a gas and/or of a partial vapour and/or of an aerosol, then the fission products can react, *in statu nascendi*, with these components and, in such a case, these reaction products can also react with further reactants.
- As reactive substances, there can be used, for example, oxygen, nitric oxide, nitrogen dioxide, halogens, sulphur dioxide or carbonyl fluoride.
- Further reactive substances include chemical compounds which can also contain double bonds, for example, amines, alcohols, phenols, carboxylic acids, sulphonic acids, hydrogen halides, ammonia and vinyl compounds. Reactive substances also include polymers, for example, poly-amides, polyesters, polyvinyl compounds, polyacrylonitrile and polystyrene. The reactive substances can also be polymers already modified with a monomer, for example with acrylic acid or acrylamide, by, for example, radiation chemical graft copolymerisation, whereby special properties can be achieved due to the combination of the individual properties of the reaction components.
- The ionising radiations used are preferably electron beams which can have an energy of 0.7 to 2 MeV.
- It is also advantageous that in a heatable irradiation vessel a substance to be decomposed and/or reacted with a reactive substance is adjusted by means of a layer thickness adjuster to about the maximum penetration depth of the electron radiation used and distributed over a heatable sieve base and the excess polymer is discarded into a collection container, and a current of carrier gas is forced or drawn through the substance to be decomposed and/or to be reacted with a substance capable of reaction therewith, as well as through the heat-adjustable sieve base, the volatile products formed being passed through a condensation device and fractionated and the liquid products are withdrawn from a collection device.
- It is advantageous that the irradiation of the fluorinated hydrocarbon polymers, especially of polytetrafluoroethylene, is undertaken from below a suspended vessel, the material to be irradiated trickling between cooling lamellae counter to the irradiation beam. The volatile reaction products are led off by means of a current of carrier gas of appropriate temperature and the reaction products of low volatility flow off into a collection vessel.
- Furthermore, by alteration of the number and the temperature of the cooling lamellae, of the temperature of the cooling medium, of the temperature of the current of carrier gas and of the radiation capacity of the electron beam, the chain length range of the products can be controlled in the condensation device, as well as in the collection vessel.
- The following Examples are given for the purpose of illustrating the present invention, reference thereby being made to the accompanying drawings which schematically illustrate three different types of apparatus for carrying out the process according to the present invention:—
- Example 1 (Fig. 1),
- Polytetrafluoroethylene or polytetrafluoroethylene waste is placed in a layer in a double-walled irradiation vessel (1) with temperature-controlling means (2) up to a height which is substantially greater than the maximum range of an electron beam (3) into the material to be irradiated (4). By means of this arrangement, there is achieved the complete utilisation of the capacity irradiated in, which hitherto has not been successful in the case of radiation-chemical reactions carried out on a large scale. The upper layer, which corresponds to the "useful range" of the irradiations, is broken down, whereas the underlying layer, extending to the maximum range, is already pre-irradiated. During the irradiation, a continuous current of nitrogen is passed through a carrier gas inlet (5) and outlet (6), over the material (4) to be irradiated. Depending upon the temperature, which can be varied within wide limits by the absorbed dosage per time unit and the temperature controlling means of the double-walled vessel, the products formed which are volatile at the temperature in question are led away, together with the current of nitrogen passed over the irradiated material, from the irradiation zone and fractionally condensed in a connected cooling system (7). A thermal insulation (8) directly connected on to the irradiation vessel (1) prevents premature

condensation of the highest boiling (longest chained) fission products. The lower boiling and thus short chained products formed condense in the after-connected receiver or in a cold trap. The filling level of the double-walled irradiation vessel (1) is automatically regulated by means of a filling height measurement device (9), for example rubber bars, and a topping up system (10) connected therewith. The irradiation vessel (1) is sealed off with a radiation-permeable foil (11).

In the case of the action of electron beams with an energy of 1.0 MeV and with an inert carrier gas current of 10 litres/hour, at a radiation capacity of 1kW and in a period of 1 hour, 36 g. polytetrafluoroethylene can be converted into a low molecular weight condensate. This condensate consists of *n*-perfluoroparaffins and straight-chain perfluorinated olefins.

Example 2.

Polytetrafluoroethylene is treated in the manner described in Example 1, the inert carrier gas being replaced by air. Using the same temperature, radiation capacity and radiation energy, the amount of polytetrafluoroethylene decomposed increases to 450 g./hour. As reaction products, there is obtained carbonyl fluoride, together with *n*-perfluorocarboxylic acid fluorides. The *n*-perfluorocarboxylic acid fluorides obtained are not stable and are hydrolysed by atmospheric moisture to give the corresponding *n*-perfluorocarboxylic acids.

Example 3.

In the case of the action of electron beams with an energy of 1.2 MeV, with an oxygen flow-through of 60 litres/hour and at a radiation capacity of 600 W, 200 g./hour of a copolymer of tetrafluoroethylene with 10 mol % hexafluoropropylene are decomposed.

Example 4.

In the case of the action of electron beams with an energy of 1.2 MeV on polytetrafluoroethylene turnings, an oxygen flow-through rate of 40 litres/hour and a radiation capacity of 600 W, there are formed, per hour, 250 g. of low molecular weight perfluorinated fission products. Fission products of this type are of especial interest for the further production of active intermediate products with perfluorinated character, for example, for the production of perfluorinated carboxylic acids.

By means of the process according to the present invention, the irradiation can be carried out continuously and without maintenance, the radiation capacity being completely utilised. As a result of the fractional condensation, fission products are

obtained with a relatively narrow range of carbon atoms in various fractions.

Example 5.

At a temperature of 180°C. and with an ammonia content of 30% in the current of carrier gas, about 400 g./hour polytetrafluoroethylene are decomposed. The condensate collected in the condensation system contains, in addition to carbon and fluorine, 1.8% nitrogen and 0.3% hydrogen.

In the following Examples, the energy of the electron beams is 1.0 MeV at a radiation capacity of 1 W. The temperature of the dry carrier gas current used is, on average, 180°C.

Example 6.

When using polytetrafluoroethylene as starting material and a current of carrier gas containing 10% nitrogen monoxide, the condensate formed contains, in addition to the elements fluorine and carbon, on average, 1.8% nitrogen and 2.0% oxygen.

Example 7.

When using polytetrafluoroethylene as starting material and a current of carrier gas containing sulphuryl chloride, condensates are formed which contain, in addition to sulphur hexafluoride, various sulphur- and chlorine-containing compounds. In addition to the elements fluorine and carbon, there can be detected in the reaction product, on average, 3.5% chlorine, 3.4% sulphur and 3.2% oxygen.

Example 8.

Outside of the irradiation zone, sulphur dichloride is sprayed counter to products which preferably result by the irradiation of polytetrafluoroethylene. Besides sulphur hexafluoride, various sulphur- and chlorine-containing condensates are thereby formed. In addition to the elements fluorine and carbon, on average there could be detected in the intermediate product 4% chlorine and 1.8% sulphur.

Example 9.

The decomposition products of polytetrafluoroethylene are contacted, *in statu nascendi*, with polymerisable substances, for example with vinyl compounds. The copolymers which thereby result combine, in part, the properties of polytetrafluoroethylene with those of the other polymer or even exhibit completely novel properties.

Example 10.

A copolymer of 90 mol % tetrafluoroethylene and 10 mol % hexafluoropropene is decomposed and the fraction of the products boiling between 100 and 140°C. and subsequently activated with electron beams is brought into contact with

polystyrene. The reaction which thereby takes place leads to the grafting of the polystyrene.

Example 11

The products formed by the action of electron beams upon a copolymer of 90 mol % tetrafluoroethylene and 10 mol % hexafluoropropene are brought into contact with a polyester film in the irradiation field, grafting thereby taking place. The graft yield can be controlled by the residence time of the polyester in the irradiation and decomposition chamber.

Example 12.

A textile planar body made from polyamide fibre materials is subjected to the same conditions as the polyester film in Example 11, the irradiated starting material being polytetrafluoroethylene. The irradiation dose absorbed in the polyamide is 5 Mrad. A product is obtained grafted with the decomposition products of polytetrafluoroethylene, this product being suitable for various textile uses.

Example 13 (Fig. 2).

Polytetrafluoroethylene (4) to be decomposed and/or reacted with a reactant is piled on to a sieve base (12) of a radiation vessel (1). The temperature-controllable sieve base (12) consists, for example, of electrically-heatable resistance wires. The top of the irradiation vessel is covered by a radiation-permeable foil (11), for example an aluminium foil. The bed height of the substance (4) is precisely adjusted by means of a rake (13) or similar device. From a supply container (14), the substance to be decomposed and/or to be reacted with a reactant is uniformly distributed, with the help of the rake (13), on the sieve base (12) and excess substance (4) is discarded into a collection container (15) and from there returned to the supply container (14). With the help of a heatable current of carrier gas entering at (5), which can be regulated with regard to the amount flowing through, to which can, if desired, be admixed appropriate reactants, and which is forced or drawn through the irradiation vessel (1), the products formed which are volatile under the selected irradiation conditions, are passed through a condensation device (17) and there fractionally condensed, the carrier gas emerging at (6). The liquid products formed by the selected irradiation conditions drop into an exchangeable collecting device (18) where, because of the complete absorption of the electron radiation (3) in the substance layer (4) to be decomposed and/or reacted with a reactant, are no longer impinged upon by the electron beam (3). The chain length of the resultant

gaseous and liquid products is influenced especially by the selected dosage capacity (beam current), as well as by the temperature and the rate of flow of the carrier gas current. The bed height of the substance (4) to be decomposed and/or reacted with a reactant corresponds precisely to the maximum range of the electron beam employed; it can be varied within relatively wide limits by the energy of the electron radiation.

This device has the decisive advantage that the carrier gas current is passed through the layer of substance, whereby, on the one hand, the conversion is increased and, on the other hand, a cooling of the substance heated by the absorption of energy is achieved. The substance to be decomposed and/or to be reacted with the help of a reactant is preferably used in the form of small turnings or granulate, in order to achieve the highest possible specific surface area.

Example 14 (Fig. 3).

A suspended vessel (19), comprising a cooling mantle (20), cooling lamellae (21) for the material (4) to be irradiated and a sieve base (12) upon which the material to be irradiated is added from above. The resultant readily volatile reaction products are carried away by a carrier gas current which enters at (5), passes under the material to be irradiated and/or can be passed from above through the material to be irradiated, removed from the irradiation zone through (6) and collected in a condensation device (17). The sparingly volatile products formed which are liquid at the selected reaction conditions, drop through the sieve base (12) on the oblique irradiation window (22) and flow into the collection vessel (18). Depending upon the temperature of the cooling medium in the cooling lamellae (21), the temperature of the carrier gas current introduced through inlet (5) and of the beam current of the electron irradiation (3), the chain length range of the products in the condensation device (17), as well as in the collection vessel (18), can be controlled.

This device has the special advantage that the decomposition of polytetrafluoroethylene or of analogous fluorinated hydrocarbon polymers can be carried out at low temperatures so that thermal decomposition and the formation of thermal decomposition products is avoided.

In our co-pending Patent Application No. 53330/74 (Serial No. 1,496,347), there is described and claimed a process for the decomposition of polymers and/or oligomers by irradiation with ionising rays and optional reaction of the intermediately formed fission products with reactants

which react, under the selected conditions, with the fission products, wherein polymers and/or oligomers of different molecular weight are irradiated in a turbulent liquid bed, one or more components of the irradiated material being liquid under the selected conditions and the decomposition and the optional reaction being carried out in the presence of one or more gas currents, the products obtained having substantially uniform chain lengths.

WHAT WE CLAIM IS:—

1. Process for the production of perfluoro hydrocarbons with a chain length of up to 30 carbon atoms or of substituted derivatives thereof or of corresponding perfluorocarboxylic acids from solid perfluorinated hydrocarbon polymers, wherein a solid perfluorinated hydrocarbon polymer is irradiated with electron beams, the intermediately formed fission products are optionally reacted with a reactant and the perfluoro hydrocarbons or substituted derivatives thereof or the corresponding perfluorocarboxylic acids thereby formed are immediately removed from the irradiation zone.
2. Process according to claim 1, wherein the irradiated material is polytetrafluoroethylene.
3. Process according to claim 1 or 2, wherein substances which are reactive under the selected radiation conditions are also passed in.
4. Process according to any of the preceding claims, wherein the reaction products are immediately removed from the irradiation zone by a current of carrier gas.
5. Process according to any of the preceding claims, wherein the reaction products obtained, optionally in the presence of materials which promote the decomposition, are brought into contact with and reacted with a substance capable of reaction with the irradiation products.
6. Process according to claim 4 or 5, wherein the irradiated material and/or the carrier gas current are used at an elevated temperature of up to the temperature of thermal decomposition of the polymer starting material.
7. Process according to any of the preceding claims, wherein the irradiated material is supplemented by fresh polymer in an amount corresponding to the amount of polymer which has been converted.
8. Process according to any of the preceding claims, wherein a bed of predetermined thickness of material to be irradiated is passed under a source of radiation and unreacted or partially reacted material is recovered and/or recycled.
9. Process according to any of claims 4 to 8, wherein the carrier gas used is one which

becomes reactive during the course of the process.

10. Process according to claim 9, wherein the carrier gas used is oxygen or a mixture of oxygen and inert gas.

11. Process according to any of the preceding claims, wherein the products formed are fractionally condensed and/or selectively separated from the carrier gas.

12. Process according to any of the preceding claims, wherein a substance which becomes reactive during the course of the process is added to the solid polymer before the commencement of the irradiation.

13. Process according to any of claims 4 to 12, wherein a substance which becomes reactive during the course of the process is added to the carrier gas current in the form of a gas and/or of a partial vapour and/or of an aerosol.

14. Process according to any of the preceding claims, wherein the reaction is carried out continuously.

15. Process according to any of claims 1 to 13, wherein the reaction is carried out discontinuously.

16. Process according to any of claims 1 to 15, wherein decomposition products, after deactivation thereof, are again treated with electron beams and brought into contact with and reacted with a substance capable of reaction therewith.

18. Process according to any of the preceding claims, wherein the products formed are first fractionated and subsequently reacted with a substance capable of reaction therewith, as individual fractions or in groups of fractions.

19. Process according to any of the preceding claims, wherein the products formed are removed from the irradiation zone with the help of a carrier gas current, separated by fractional condensation and the individual fractions are reacted with a substance capable of reaction therewith.

20. Process according to claim 18, wherein the substance capable of reaction is an amine, alcohol, phenol, carboxylic acid, sulphonic acid, hydrogen halide, vinyl compound or ammonia.

21. Process according to any of the preceding claims, wherein, in a temperature-controllable irradiation vessel, the polymer or oligomer to be decomposed and/or to be reacted with a substance capable of reaction therewith is adjusted by means of a layer thickness adjuster to about the maximum penetration depth of the electron beam used and distributed over a temperature-controllable sieve base, excess polymer being discarded into a collection container, a carrier gas current is forced or drawn through the polymer or oligomer to be decomposed and/or reacted with a

- substance capable of reaction therewith, as well as through the sieve base, the volatile products formed or the products obtained by reaction with a substance capable of reaction therewith are passed through a condensation device and fractionated and the liquid products are withdrawn from a collection device.
21. Process according to any of claims 1 to 19, wherein, in a suspended vessel, the material to be irradiated is passed counter to the irradiation beam through cooling lamellae, irradiated from below and the resultant decomposition products are passed by the carrier gas current into a condensation system or passed directly into a collection vessel.
22. Process according to claim 21, wherein the number and the temperature of the cooling lamellae, the temperature of the cooling medium, the temperature of the carrier gas current and the radiation capacity of the electron beam are varied.
23. Process according to any of the preceding claims, whenever carried out in a device comprising a temperature-controllable, double-walled irradiation vessel for containing the material to be irradiated, a gas inlet and gas outlet arranged at the height of the reaction zone, a sealing and irradiation-permeable foil and means for regulating the electron density or the degree of decomposition in the irradiation bed.
24. Process according to claim 23, wherein the irradiation vessel of the device has a temperature-controllable sieve base below which is arranged a collecting device.
25. Process according to claim 24, wherein a transparent window is present below the sieve base.
26. Process according to any of the preceding claims, whenever carried out in a device substantially as hereinbefore described and exemplified and with reference to the accompanying drawings.
27. A process according to claim 1, for the production of perfluoro-hydrocarbons and substituted derivatives thereof, as well as of the corresponding perfluorocarboxylic acids, substantially as hereinbefore described and exemplified.
28. Perfluoro-hydrocarbons and substituted derivatives thereof, as well as the corresponding perfluorocarboxylic acids, whenever produced by the process according to any of claims 1 to 27.

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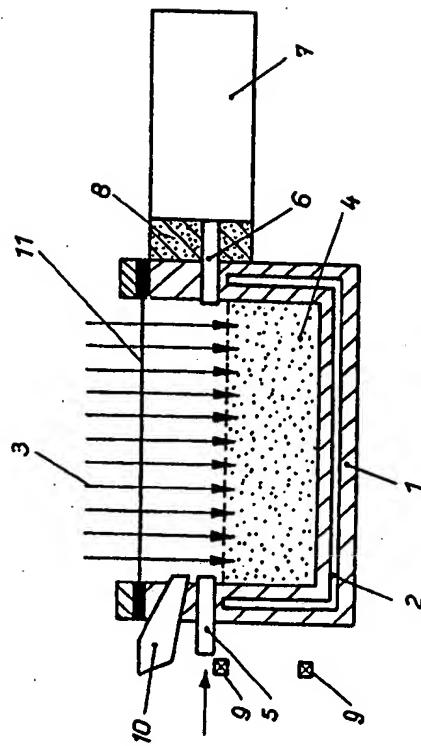


Fig. 1

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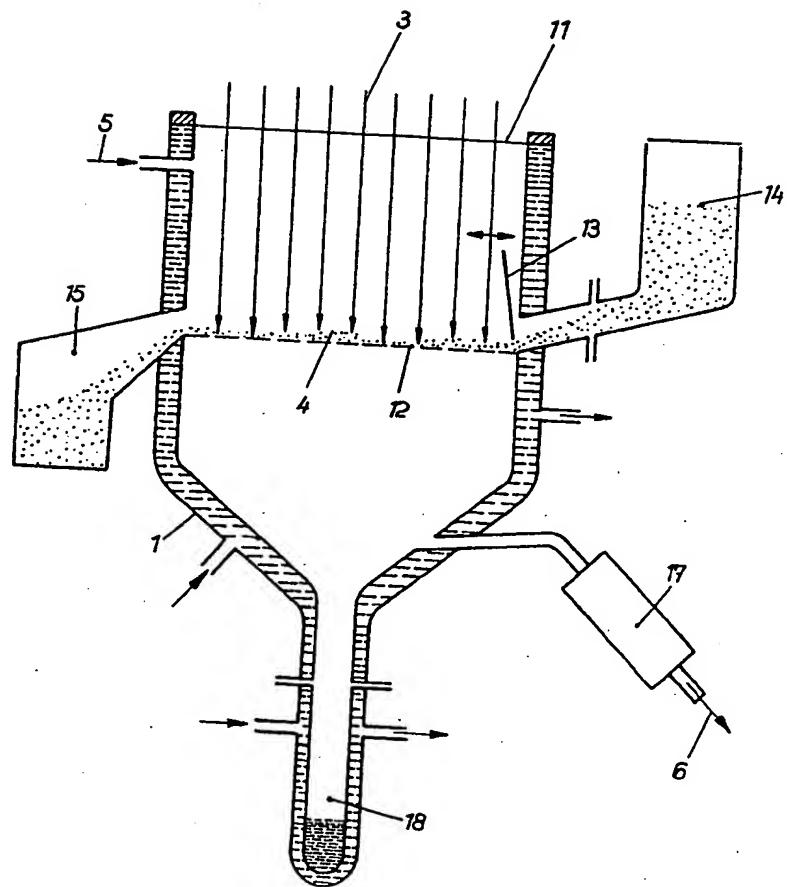


Fig. 2

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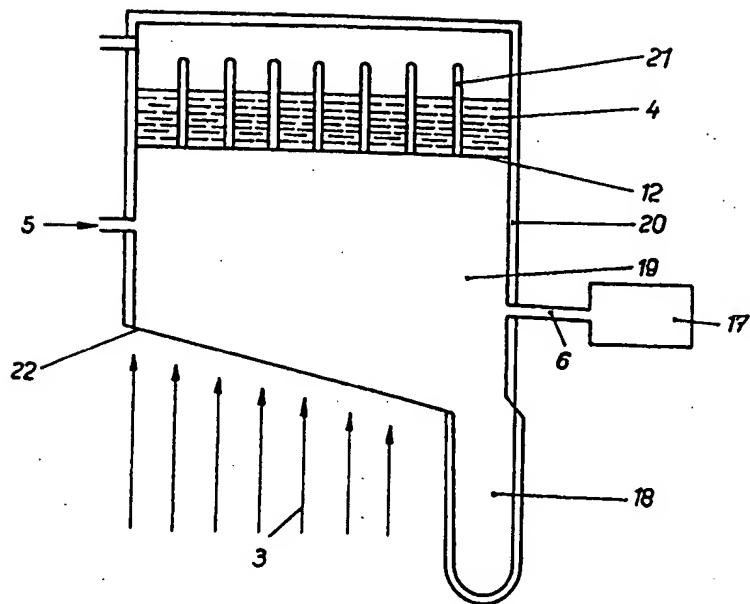


Fig. 3